



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: :
Andrzej Rokicki, et al. :
:
Serial No.: 10/047,598 :
: Group No. 1754
Filed: January 14, 2002 :
: Examiner: Stuart L.
Attorney Docket No.: P-1115 :
: Hendrickson
For: DEHYDROGENATION CATALYST :
: Confirmation No. 5752
:
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AFFIDAVIT UNDER 37 CFR 1.132

Vladimir Fridman, being duly sworn, does hereby depose and state as follows:

1. That I received a Doctor degree in Chemistry from Moscow University on the 8 day of May, 1992.

2. I am co-inventor of patent application Serial No. 10/047,598, filed on January 4, 2002, entitled "DEHYDROGENATION CATALYST."

3. That I have been employed by Süd-Chemie, Inc., in Louisville, Kentucky since July 10, 2000, and that my position at Süd-Chemie, Inc. is Group Leader.

4. There are different types of alumina that may be used as a support for catalysts. However, mid to high surface area, gamma alumina has consistently been the preferred choice as the carrier

for dehydrogenation catalysts, as disclosed, for example, in U.S. Patent Nos. 2,956,030, 2,945,823 and 2,374,404.

In particular, gamma alumina is preferred over eta alumina as the carrier material for dehydrogenation catalysts. For example, in Tsuchida, et al., "The effect of Cr³⁺ and Fe³⁺ ions on the transformation of different aluminum hydroxide to alpha-Al₂O₃", *Thermochimica ACTA*, 64, pages 337-353 (1983), the preference for gamma alumina over eta alumina is clear. The article states that during the formation of alpha alumina containing Cr³⁺ ions, the transformation of bayerite containing chromium ions from eta alumina to alpha aluminum was "accelerated." In contrast, the transformation of boehmite containing chromium ions from gamma alumina to alpha alumina was "inhibited." Acceleration of this transformation to alpha alumina, as is exhibited by eta alumina, results in reduced stabilization of the catalyst while inhibition in the transformation, as is exhibited by gamma alumina, enhances stabilization of the catalyst end product.

The preference for gamma alumina as the support material for catalysts in general, especially where enhanced stability at higher temperatures is required, is also discussed in Richardson, James T.; *Principles of Catalyst Development*, (1989). The preference for gamma aluminum as a support material is specifically discussed at pages 35 and 36, especially in a situation where a small quantity of zirconia is added to the alumina to stabilize the catalyst.

Another example of the preference for gamma alumina over eta alumina as the material used to form the carrier of a dehydrogenation catalyst is disclosed in U.S. Patent No. 2,943,067. At column 5, Example 1, the performance of an alumina supported catalyst produced by the Bayer process (which produces a gamma alumina) is described as being superior to a catalyst prepared from a gel-type alumina, which upon heating normally converts to an eta alumina. (Alumina produced by the Bayer process produces gibbsite, which upon heating converts to gamma alumina.) Thus, the '067 patent teaches the superiority of gamma alumina over eta alumina as the carrier for dehydrogenation catalysts.

The lack of thermal stability for catalyst produced from eta alumina is also discussed in Oberlander, Richard K.: *Aluminas for Catalysts - Their Preparation and Properties*, page 69 (1983).

This preference for gamma alumina over eta alumina as the carrier for dehydrogenation catalysts is not surprising because gamma alumina is generally perceived as having a greater thermal stability than eta alumina. In fact, gamma alumina has become the standard alumina utilized for dehydrogenation catalysts. (The market has accepted this principle as gamma alumina is readily available in the market while eta alumina is sparsely available, if at all.)

Although dehydrogenation catalysts prepared from chromia-alumina catalysts have been extensively employed for many years,

there are still problems with current catalysts, especially their thermal stability. Even when these catalysts are stabilized by the addition of an additive, such as by addition of a zirconium or a silicon compound, these catalysts still show limited stability because of the severity of the operating conditions, particularly the high temperatures that occur during the dehydrogenation procedure.

5. I have surprisingly discovered that dehydrogenation catalysts made from a combination of an eta alumina impregnated with chromia and zirconia, as a stabilizer, provide improved performance over prior art dehydrogenation catalysts, particularly gamma alumina- based dehydrogenation catalysts.

6. In order to establish this enhanced performance, I have run examples where I have compared the performance of dehydrogenation catalyst prepared using conventional gamma alumina carriers with similar dehydrogenation catalyst using eta alumina carriers of the invention. In these examples, the composition of the catalyst is as follows:

<u>Examples</u>	<u>Composition</u>
Example 1 (Ex.1)	19% Cr_2O_3 , remaining portion eta alumina
Example 2 (Ex.2)	19% CrO_2 , 0.7% ZrO_2 , remaining portion eta alumina
Comparative Example 3 (CEX.3)	19% Cr_2O_3 remaining portion gamma alumina
Comparative Example 4 (CEX.4)	19% CrO_2 , 0.7% ZrO_2 , remaining portion gamma alumina

7. The performance of these catalysts was tested for isobutane dehydrogenation performance in an externally heated tubular reactor of one inch inner diameter. Isobutane was introduced to the catalyst at a controlled throughput and pressure (LHSV=1.02 and pressure = 0.33 atm.) over a range of temperatures from 537°C to 593°C. Dehydrogenated products were analyzed to determine conversion of isobutane, selectivity to isobutylene, yield of isobutylene, yield of light gas C₁ to C₃ and yield of coke. In order to test the catalysts as aged, each of the catalysts of the examples was artificially aged at the same conditions at high temperature in a tubular reactor of 2 inch internal diameter. To provide various levels of the catalyst age, different dehydrogenation temperatures were used. The activity of the artificially aged catalysts is shown as follows:

Performance Tests	(CEx. 3)	(CEx. 4)	(Ex. 1)	(Ex. 2)
1000°F (dehydrogenation temp.)				
C1 to C3:[wt%]	1.2	1.6	0.8	1.1
Isobutane Conversion:[wt%]	27.0	39.3	48.2	59.7
Isobutylene Selectivity:[wt%]	89.3	89.8	92.2	92.3
Isobutylene Yield:[wt%]	23.8	35.1	44.5	55.0
Coke Yield [wt%]	0.7	0.7	0.6	0.7
1050°F (dehydrogenation temp.)				
C1 to C3:[wt%]	2.9	3.5	3.3	3.2
Isobutane Conversion:[wt%]	44.1	50.9	65.8	72.1
Isobutylene Selectivity:[wt%]	84.7	87.1	85.3	88.0
Isobutylene Yield:[wt%]	37.4	44.4	56.1	63.4
Coke Yield [wt%]	1.5	0.6	2.5	1.4

1100°F				
C1 to C3:[wt%]	7.7	9.8	7.2	8.1
Isobutane Conversion:[wt%]	59.5	65.7	74.1	80.0
Isobutylene Selectivity:[wt%]	72.1	77.1	74.4	77.8
Isobutylene Yield:[wt%]	42.9	50.7	55.1	62.2
Coke Yield [wt%]	5.3	1.8	6.7	4.6

8. As is clear from these Examples, the performance of the aged catalyst of the invention using eta alumina as the carrier is surprisingly better than the performance of a conventional dehydrogenation catalyst produced from gamma alumina-chromia stabilized with zirconium. This was a surprising improvement, especially based on the teaching of the prior art that gamma alumina based catalyst out-preformed eta alumina-based catalyst.

Further, affiant saith not.

Vladimir Fridman

Vladimir Fridman

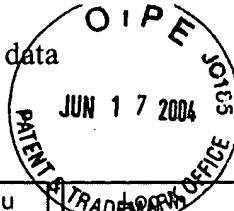
STATE OF KENTUCKY)
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Acknowledged, subscribed and sworn to before me this 11th day of June, 2004, by Vladimir Fridman to be his act and deed.

My commission expires: Aug 11 2004

Notary Public, State at Large, KY

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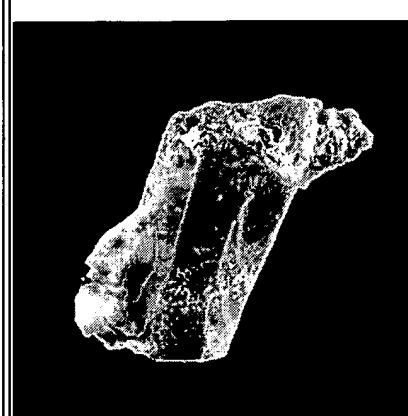
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Corundum



Limecrest Quarry (Crestmore Quarry),
Sparta Township, Sussex Co., New Jersey,
USA

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Show Locs (276) Corundum Gallery

Formula: Al_2O_3

System: Trigonal **Colour:** Colourless, blue, red, ...

Hardness: 9

Name: Derived from the Sanskrit, kuruvinda
("Ruby")

This page provides mineralogical data about
Corundum.

Classification of Corundum

IMA status:	Approved
Validity of Species:	A valid mineral species
Strunz ID:	4/C.04-10 4 : Oxides & Hydroxides C : Oxides with metal : oxygen = 2:3 (M ₂ O ₃ and related compounds) 04 : Hematite group
Dana ID:	4.3.1.1
Hey's CIM Ref.:	7.6.1
mindat.org URL:	http://www.mindat.org/min-1136.html Please feel free to link to this page.

Occurrences of Corundum

Geological Setting:	Silica-poor rocks, such as Nephelene-Syenites, alkali igneous undersaturated rocks, contact aureoles in altered aluminous shales, aluminous xenoliths in high temperature plutonic and hypabyssal rocks, metamorphosed bauxite deposits, and as a detrital material in sediments.
Industrial Uses:	Corundum is used as an abrasive ("Emery"), and the gem varieties of corundum are better-known as Ruby and Sapphire.

Physical Properties of Corundum

Lustre:	Vitreous
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Diapheny:	Transparent, Translucent
Colour:	Colourless, blue, red, pink, yellow, grey
Streak:	White
Hardness (Mohs')	9
Hardness Data:	Mohs' hardness reference species
Cleavage:	None Observed
Fracture:	Irregular/Uneven
Density (measured):	4 - 4.1 g/cm ³
Density (calculated):	4.01 g/cm ³

Crystallography of Corundum

Crystal System:	Trigonal
Class (H-M):	$\bar{3}$ 2/m - Hexagonal Scalenohedral
Space Group:	$\bar{R}\bar{3}c$
Cell Parameters:	$a = 4.751, c = 12.97$
Ratio:	$a:c = 1 : 2.73$
Unit Cell Volume:	$V = 253.54 \text{ \AA}^3$
Z:	6
Morphology:	Euhedral; small rounded crystals; alluvial crystals and grains

Optical Data of Corundum

Type:	Uniaxial (-)
RI values	$n_w = 1.768 - 1.772$ $n_e = 1.760 - 1.763$
Maximum Birefringence:	$\delta = 0.008 - 0.009$ 
	Chart shows birefringence interference colour range (at 30µm thickness) and does not take into account mineral colouration.
Surface Relief:	High
Colour in reflected light:	Colourless

Relationship of Corundum to other Species

Related Minerals (Strunz Grouping):	4/C.04-20 Hematite Fe_2O_3 4/C.04-30 Eskolaite Cr_2O_3 4/C.04-40 Karelianite V_2O_3
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Chemical Properties of Corundum

Formula:	Al_2O_3
Elements:	Al, O
Analytical Data:	Al (52.93) O (47.07)
Empirical Formula:	Al_2O_3
Common Impurities:	Cr, Fe, V, Ti

Other Names for Corundum

Synonyms:	<ul style="list-style-type: none"> • α-Alumina • Adamas siderites • Ayatit • Corindon • Corivendum • Corundit • Corundum-a • Corundumita • Korund • Korunduvite • Soimontite • Zircolita • α-Corundum • Ajatit • Ayatita • Corinendum • Corivindum • Corundita • Corundum-alpha • Corundumite • Korunduvit • Soimontit • White Sapphire • Zirconite • a-Corundum • Alumina • Ayatite • Corinindum • Corrindon • Corundite • Corundumit • Harmophane • Korunduvita • Soimontita • Zircolit • Adamant (in part)
Varieties:	<ul style="list-style-type: none"> • Adamantine Spar • Chlorosapphir • Oriental Emerald • Ruby • Alundum • Ledo frozen fire • Oriental Topaz • Sapphire • Blue Alexandrite • Oriental Amethyst • Padmaragaya

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